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Evaluation of Sensitized Fluorescence for Polynuclear Aromatic Hydrocarbon Detection

TRW Defense and Space Systems Group, Redondo Beach, CA

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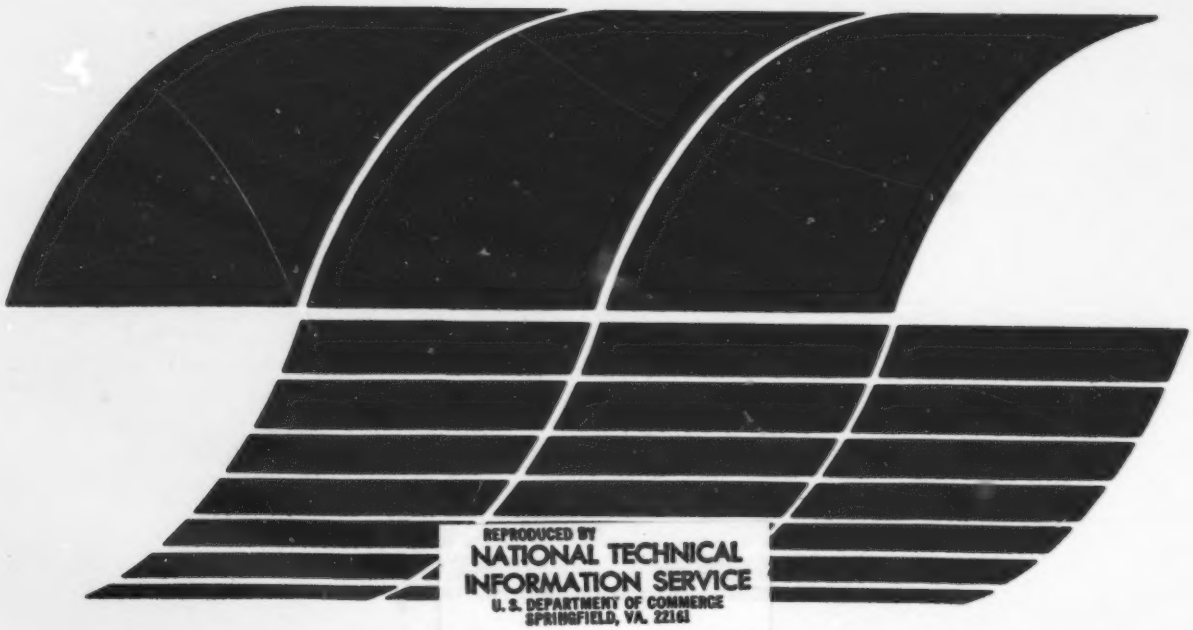

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16. ABSTRACT The report gives results of an evaluation of a fluorescent spot test for detecting the presence of polynuclear aromatic hydrocarbons (PAHs) as a screening technique for samples to be analyzed by Gas Chromatography/Mass Spectrometry (GC/MS). The test is based on the phenomenon of sensitized fluorescence and is capable of easily detecting 100 picograms of PAH in a 1 microliter sample, a level of sensitivity adequate for screening combustion effluent samples. Two interferences were observed: highly colored samples require dilution to allow viewing of the fluorescence level, and samples containing substantial amounts of phthalate esters produce false positive results. No false negative results were observed in the study. The test is adequate for screening combustion effluent samples prior to GC/MS analysis.

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Combustion Products	Stationary Sources	21B
Screening	Sensitized Fluorescence	14B
Aromatic Polycyclic Hydrocarbons		07C
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by

T. R. Smith

TRW Defence and Space Systems Group
One Space Park
Redondo Beach, California 90278

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EPA Project Officer: Larry D. Johnson

Industrial Environmental Research Laboratory
Office of Energy, Minerals, and Industry
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Abstract

A flourescent spot test used for detecting the presence of polynuclear aromatic hydrocarbons (PAHs) has been evaluated as a screening technique for samples to be analyzed by gas chromatography/mass spectrometry (GC/MS). The spot test is based on the phenomenon of sensitized fluorescence and is capable of easily detecting 100 picograms of PAH in a 1 microliter sample, a level of sensitivity adequate for the screening of combustion effluent samples.

Two interferences were observed: 1) Samples which are highly colored required dilution to allow viewing of the fluorescence level and 2) samples containing substantial amounts of phthalate esters produced false positive results. No false negative results were observed in this study.

It is our conclusion that the spot test procedure is adequate for the screening of combustion effluent samples prior to GC/MS analysis.

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1.0 INTRODUCTION

This document represents the final report on the evaluation of the Polynuclear Aromatic Hydrocarbon Spot Test. This report is submitted in partial fulfillment of the requirements for EPA contract No. 68-02-2689, Technical Directive 104. The work was conducted by TRW under the direction of EPA Task and Project Officer, Dr. L. D. Johnson.

The original fluorescence spot test discussed in this report was developed by Arthur D. Little, Inc.[1] under contract to the EPA and is based on the phenomenon of sensitized fluorescence. The objective of this effort was to determine if the test could be used as a screening aid for samples submitted for PAH analysis by gas chromatography/mass spectrometry (GC/MS). The procedure can potentially provide a low cost screening technique to predetermine the presence of PAHs.

This test was evaluated specifically for its detection of PAHs. The importance of PAHs as a class originate from the serious health hazard posed by certain components of this class of compounds. Specifically, benzo(a)pyrene and dibenzo(a,i)pyrene are compounds within this class which have been shown to be potent cancer causing agents. The potential danger of contracting cancer is incurred when these compounds are inhaled into the lungs or come into contact with the skin. Therefore, it is important that these compounds be detected and identified in effluents which could come into contact with the population.

The analysis schemes thus far implemented for the determination of PAHs encompass several analytical approaches including nonspecific analysis, detailed quantitative and qualitative analysis, and specific analysis for a single PAH. The limits of detection have been reported to be at the 10 picogram level in some instances. The analysis times range from 15 minutes to several hours with the evaluation if the test results occupying an equal amount of time. The PAH spot test is a survey technique which provides information as to the presence or absence of PAH as a class and no specificity is provided. The level of detection is in the picogram range and the analysis time is on the order of 15 minutes. Gas chromatography/mass spectrometry can provide qualitative and quantitative information on the PAHs present in an effluent. Sample

analysis time is typically between 1 to 2 hours with the data evaluation consuming at least an equal amount of time. The limits of detection for the GC/MS analysis normally approaches 5 nanograms per PAH.

For general purpose determination of PAH in complex mixtures the use of GC/MS is unexcelled. The gas chromatograph is used to separate the complex mixture into individual component peaks and the mass spectrometer is used as a detector for both qualitative identification of the PAH and for quantitative determination. For simple cases where only a few PAH are present, the use of packed gas chromatographic columns is recommended. Packed columns are generally easier to use, require a shorter time for analysis and are cheaper than capillary columns. When highly complex mixtures of PAH are present or significant interferences occur, the use of capillary columns becomes necessary. Chromatographic column selection is usually dependent on the volatility and polarity of the PAH to be determined.

The major limitation of the gas chromatograph is the volatility of compounds to be analyzed. In order to be determined by GC/MS, PAHs must produce good chromatographic peaks at temperatures below 300°C. At or above 300°C even the most stable GC column liquid phases produce high background in the mass spectrometer. This GC temperature limitation means that high molecular weight PAHs (above molecular weight 300) cannot be reliably determined by this technique. For this reason some PAHs which produce fluorescence in the spot test may not be analyzable by GC/MS.

As discussed above, the analysis of samples by GC/MS for the routine detection of PAHs expends costly GC/MS instrument time as well as the time needed to interpret the data. The PAH spot test is proposed to serve as a screening tool for the GC/MS analyst. This pretest will serve as a go/no go analysis. That is, if the test indicates the presence of PAH, the sample will be run via GC/MS. If no fluorescence is observed it will not require analysis. Since the PAH spot test requires only about 15 minutes of effort and no instrument time, implementation of this test should result in a significant decrease in PAH analysis time and cost. Clearly, this is contingent upon several factors. First, for maximum benefit to the combustion environmental assessment program sensitivity should equal or exceed that of GC/MS for other projects the sensitivity

requirement varies with the job. Second, interferences which would result in false negative findings must be either identified and taken into consideration or they must be nonexistant. The experiments reported here were designed to evaluate these possibilities.

2.0 EXPERIMENTAL CONDITIONS

Phenanthrene and benzo(a)pyrene were chosen as model analytes to test the spot test procedure. This choice was based on the facts that:

- Phenanthrene and benzo(a)pyrene are representative of PAHs which are likely to occur in combustion samples.
- The range of aromatic fused ring compounds detectable by GC/MS is covered by these two compounds.
- Any difference in sensitivity of the spot test to PAHs should be discernable by the comparison of the response of these two components,
- and lastly, because of benzo(a)pyrene's carcinogenicity, it is critical that the presence of this compound be detected by the spot test at low concentrations.

An ultraviolet Chromatovue Cabinet, Model C-70, was used for ultraviolet exposure of the samples. This unit contains both 254 and 365 nm lamps of which the 254 nm source is used for the naphthalene-PAH fluorescent detections. Whatman #42 ashless filter paper was used as a substrate for the sample analysis.

The phenanthrene and benzo(a)pyrene used for the prelimenary study were obtained from Analabs, Inc., North Haven, Conn. The solvent, methylene chloride, was obtained from Burdick and Jackson, Inc., MusKegon, MI. The sensitizer, solution, naphthalene was prepared at a concentration of 60 µg/µℓ in methylene chloride.

The gas chromatographic/mass spectrometric analyses were done on a duPont model 321 GC/MS. The key operating parameters of the GC/MS were as follows:

Column - 2 m. X 3mm ID glass packed with 3% OV-101 on 100-120 Chromosorb WHP

Carrier Gas - Zero grade helium at 30 ml/min

Oven Temperature - 100°C to 295°C programmed at 8°C/min

Injector Temperature - 300°C

Transfer lines and separator Temperature - 270°C

Mass Range - 40-450 AMU

Scan Function up - 1.9 seconds

down - 0.0 seconds

hold at top - 0.0 seconds
hold at bottom - 0.1 seconds

The high performance liquid chromatograph (HPLC) used for this report was a duPont model 850 equipped with a variable wavelength ultraviolet spectrophotometer. The wavelength monitored on all chromatograms was 254 nm. For all separations a Zorbax-ODS, 4.6 mm ID X 25 cm, liquid chromatographic column was used. All solvents were obtained from Burdick and Jackson, Inc.

The samples used to determine applicability of the spot test procedure to combustion effluent samples were provided by ongoing EPA sponsored projects. Specific sample type identifications are presented in the results section of this report.

3.0 SPOT TEST PROCEDURE

3.1 PREPARATION OF SAMPLES

No preparation of the sample is required. The sample should be in a solution to allow easy application to the test substrate. Solids and gases can be analyzed with some modification of the application technique [1].

3.2 PROCEDURE

A Chromatovue ultraviolet cabinet Model C-70 was used to expose the samples. The 254 nm lamp source was used. A 7.0 centimeter circular ashless Whatman #42 filter was used as the support for the samples. The sensitizer used was naphthalene at a concentration of 60 µg/µℓ in methylene chloride.

The spot test is carried out as follows:

1. Draw three circles in pencil on a piece of Whatman #42 filter paper as close to one another as feasible.

2. Using a 10 µℓ syringe, spot the filter paper with the sensitizer (naphthalene). 1 microliter of sensitizer should be spotted on the leftmost circle and 1 microliter of sensitizer should be spotted on the center circle. The spots should not be allowed to overlap. The substrate should be supported so as not to touch any surface.

3. Allow the solvent to evaporate from the filter paper.

4. 1 microliter of sample is then applied to the rightmost and center circles. Again allow the solvent to evaporate and do not allow the substrate to touch any surface until the solvent has evaporated.

5. The filter paper is placed in the Chromatovue cabinet.

6. Compare the spots visually with the unaided eye.

7. If the center spot (sample plus sensitizer) fluoresces brighter than the sensitizer spot (leftmost), the presence of PAHs is suspected. Also, any significant difference in color between the sensitizer only spot versus sensitizer/sample spot is also considered a positive indicator for PAHs.

8. Samples which give positive results by the spot test should be submitted for GC/MS analysis to identify the source of

the fluorescence.

4.0 DETECTION LIMIT AND SAMPLE ANALYSIS

In order to determine the level of detection attainable with the equipment available, a set of standards were prepared and tested. Phenanthrene and benzo(a)pyrene were chosen as the test standards. As previously discussed, phenanthrene and benzo(a)pyrene were chosen because both are considered representative of the PAHs likely to occur in combustion effluent samples. Their differences in structure may also cause differences in their detectability by the PAH spot test. Additionally, the range of aromatic fused ring compounds detectable by GC/MS is covered by these two compounds and the presence of benzo(a)pyrene, because of its carcinogenicity, must be detectable by the spot test at low concentrations.

The levels of standards prepared and the results of the analyses are presented in Table 1. The limit of detection of the spot test is extropolated to be approximately 100 picograms for these two materials. That is, a 1 microliter sample containing 100 picograms of PAH is easily detected. This observation differs by an order of magnitude from the detection limit of 10 picograms previously reported[1]. The difference in the reported detection limit is probably due to the point at which the investigator decides to declare a significantly distinguishable difference in the fluorescent intensity of the sensitizer alone versus the sensitizer/sample spot. Since this decision is subjective it was decided to choose a level that was easily discernable by personnel not familiar with the test. A lower detection limit is achievable but it would require the operator to make judgements based on subtleties of intensity and hue and would require extensive operator training and experience.

Another factor which must be taken into consideration when detection limits are compared is the ultraviolet lamp intensity. Any difference in lamp intensity will result in a difference of fluorescence intensity and consequently, the detectable difference between the sensitizer only and the sensitizer/sample spots would be concommitantly effected. It is an accepted fact that lamp intensity varies with age and it is not unusual to encounter significant differences in lamp intensities in an apparatus such as the Chromatovue C-70. Differences in lamp intensity as a source of variation of the detection limit was not investigated as a part of this task.

4.1 RESULTS

A series of industrial samples which gave negative results for both the PAH spot test and GC/MS analysis were spiked with a known amount of benzo(a)pyrene (100 pg/μℓ) and retested by the spot test. As expected, the samples after spiking yielded a positive indication by the PAH spot test.

Over 20 samples from industrial sites have been tested by both GC/MS and the fluorescence spot test. A partial listing of these samples and their results are given in Table 2. Approximately 10% of the samples which gave a positive result of the spot test showed no PAHs by GC/MS. As will be illustrated below, the spot test has a level of detection which is lower than that of GC/MS, when the GC/MS is used in the traditional scanning mode. Taking this fact into consideration, it is not unreasonable to expect a small percentage of the samples to prove positive by the spot test and negative by GC/MS. But what is more important is the fact that in all cases where the spot test gave negative results the GC/MS also found no PAHs. That is, no false negatives were encountered.

4.2 INTERFERENCES

The effectiveness of the PAH spot test is lessened when a highly colored sample is encountered. If the sample is opaque or highly colored, the sample must be diluted. This dilution is to allow the operator to view the fluorescence of the sample without interference from the sample's color. The detection limit of the spot test is increased proportionatly as the sample is diluted.

An example of this interference is demonstrated by the combustion effluent samples studied. It is not unusual for combustion effluent samples to posses a deep yellow color. When tested, the yellow color of the sample combined with the blue color of the naphthalene sensitizer forms a purple-blue sensitizer/sample spot. This purple-blue color of the sensitizer/sample spot is different in color from the blue sensitizer only spot. Since it has been noted that in some cases a difference in hue is an indication of PAH presence, this interference may be the source of some confusion and result in unconfirmed positives.

When the sample is highly colored, it must be diluted so that the color does not interfere with the fluorescence. For samples where color was a

Table 1

Results of spot test analysis of standards

Phenanthrene		Benzo(a)pyrene	
Amount	Test Results	Amount	Test Results
80 ng	Positive	490 ng	Positive
8 ng	Positive	49 ng	Positive
160 pg	Positive	147 pg	Positive
80 pg	Negative	98 pg	Negative
		49 pg	Negative

Table 2

Results of spot test analysis of samples from industrial sites

Sample	Spot Test	GC/MS
Coke Oven Extracts		
A	Positive	Positive
B	Positive	Positive
Combustion Effluent Samples (Commercial Oil Burner)		
A	Negative	Negative
B	Positive	Positive
C	Positive	Positive
Combustion Effluent Samples (Coal Fired Power Plant)		
A	Negative	Negative
B	Negative	Negative
C	Positive	Negative
D	Negative	Negative
E	Negative	Negative
F	Negative	Negative
G	Negative	Negative
H	Positive	Negative

source of interference, the dilution was achieved by adding more sensitizer to both the sensitizer/sample and sensitizer only spots. The sensitizer was added equally to both spots until the sample color no longer interfered. This was done to maintain the total amount of sample constant. As of this writting, no more than 3 microliters of sensitizer has been required. For samples that are highly colored, it is also suggested that the samples not only be examined immediately after application to the filter paper, but also after the sample has been exposed to the UV source for a total of 5 minutes. This delay allows for a clearer distinction between the sensitizer and sensitizer/sample spot. During this 5 minute period the fluorescence of the naphthalene diminishes and if there are PAHs present in the sample, the rate of fluorescence decay will be more rapid in the sensitizer only spot. Another suggested technique is to also view the sample under the 365 nm source. In some cases this has been found to enhance the differences in the spots.

4.3 METHODS OF IDENTIFICATION BY GC/MS

When using GC/MS polynuclear aromatic hydrocarbons are best identified as isomer classes based on molecular weight. Once a chromatographic separation has been achieved and the large quantity of mass spectral data is stored in a computer based system, individual mass chromatograms for specific molecular weights can be displayed. After it is established which classes of PAH compounds are present in a given sample, standards of the individual components or relative retention time data can be used to identify specific isomers.

PAHs typically show very strong molecular ions (the mass representative of molecular weight) in their mass spectra because of the stability of their aromatic ring system. This strong molecular ion allows for a very simple identification of molecular weight even in complex mixtures. The disadvantage of this strong molecular ion production is that isomeric PAHs produce spectra which are virtually identical. The mass spectrometer is a rather poor device for distinguishing isomers since once ionized a given compound will seek its most thermodynamically stable form. Since isomeric PAHs produce similar or identical mass spectra, the determination of structure is best accomplished by comparision of their GC retention times to standards. That is, for qualitative identification of specific

isomers, standard addition of known PAH compounds is made to each sample. An increase in GC peak intensity without broadening is used as confirmation of identification. Once a single isomer of a given molecular weight is identified, relative retention time data can be accurately applied for identification of isomers where standards are not available.

4.4 COMBUSTION EFFLUENTS (COAL BURNING POWER PLANT)

Figure 1 is an example of the total ion chromatogram of a combustion effluent sample which was analyzed by both GC/MS and the spot test. Both the GC/MS and PAH spot test yielded negative results. Examination of the mass chromatogram for m/e 252 (Figure 2) indicates that benzo(a)pyrene, perylene, etc. were not detected by GC/MS. Also, the mass chromatogram for m/e 202 (Figure 3) shows no indication of fluoranthene, pyrene, etc. This technique of using selected mass values to search GC/MS data for PAH is used routinely in our laboratory. By using mass chromatograms, GC/MS data can be rapidly searched for the most common PAHs. This technique is less time consuming than displaying the mass spectra for all of the peaks observed in a GC/MS run.

The data displayed in Figures 1-3 are typical of samples run from this combustion source. Some samples of the series gave positive results on the spot test but all proved to be negative by GC/MS.

4.5 COKE OVEN EFFLUENT SAMPLES

When coal is pyrolyzed by a burning or coking process, significant amounts of PAHs are produced. In the burning process most of the organic material is destroyed through oxidation, producing water and carbon dioxide. In the coking process, however, significant amounts of organics are evolved and emitted to the atmosphere. For these reasons the coke oven samples were an essential inclusion into the experimental scheme.

Figure 4 is a GC/MS total ion chromatogram of a typical coke oven sample. This sample was shown to contain PAHs by the spot test. Presented in Figure 5 is the mass chromatogram for the m/e 202 of this sample. In this figure, peaks containing m/e of 202 are evident. The possible identities of these peaks include the PAHs, fluoranthene and pyrene. An examination of the mass spectra of these peaks which are shown in Figure 6,

7, and 8 will more clearly aid in defining their identity. These mass spectra confirm the presence of pyrene and fluoranthene. The mass chromatogram of the 252 ion is illustrated in Figure 9. Two peaks are clearly shown which have significant abundance at m/e 252. Since the benzopyrene isomers have a molecular weight of 252 and elute in this region of the chromatogram, they serve as likely candidates. Examination of the mass spectrum of the peak eluting at scan 710 (Figure 10) confirms the presence of at least one benzopyrene isomer.

4.6 SPOT TEST VERSUS GC/MS: DETECTION LIMIT

Figure 11 is the GC/MS total ion chromatogram of a sample which gave a strongly positive PAH spot test result. The amount of sample injected onto the GC column was 3 microliters. This is 3 times the amount of sample used for the spot test. Figures 12 through 15 are the mass chromatograms for the m/e's 202, 252, 300, and 302, respectively. These represent the ions which are indicative of the PAHs typically present in these samples. In Figure 16, the area of the m/e 202 peak maximizing at scan 447 (15:00 minutes) is 53K counts. Extrapolation of this number to an area still distinguishable in the mass chromatograms suggests that the sample size could be reduced from 3.0 µl to 0.3 µl, an effective dilution of 10, and still be detectable by GC/MS. Figure 17 is the mass chromatogram of the 252 ion including the areas calculated for the components containing this ion. The previously noted extrapolation is also applicable in this case. For the purposes of comparison, this will be considered the limit of detection for the GC/MS analysis. That is, the sample, if diluted by more than a factor of 10, would not contain an amount of PAH that could clearly be detected by GC/MS.

In order to compare the aforementioned results to the detection limits of the PAH spot test, the sample was diluted and the resultant dilutions subjected to the spot test. The sample when diluted by a factor of 100 still gave a positive result by the spot test. The subsequent level of PAHs in the sample after a one hundredfold dilution was previously extrapolated to be below the detection limit of the GC/MS. Consequently, using this sample as an example, the limit of detection of the PAH spot test has been demonstrated to be more than adequate for the purpose of determining which samples are to be further analyzed by GC/MS for PAHs.

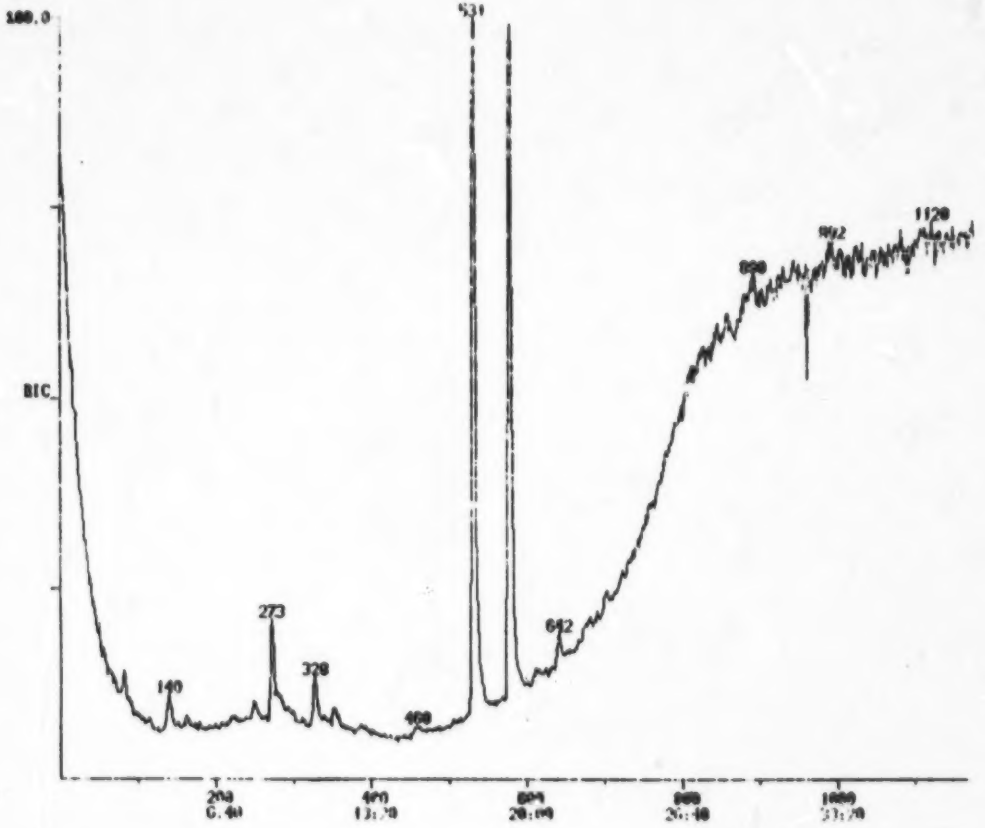

Figure 1. Total ion chromatogram of a combustion effluent sample.

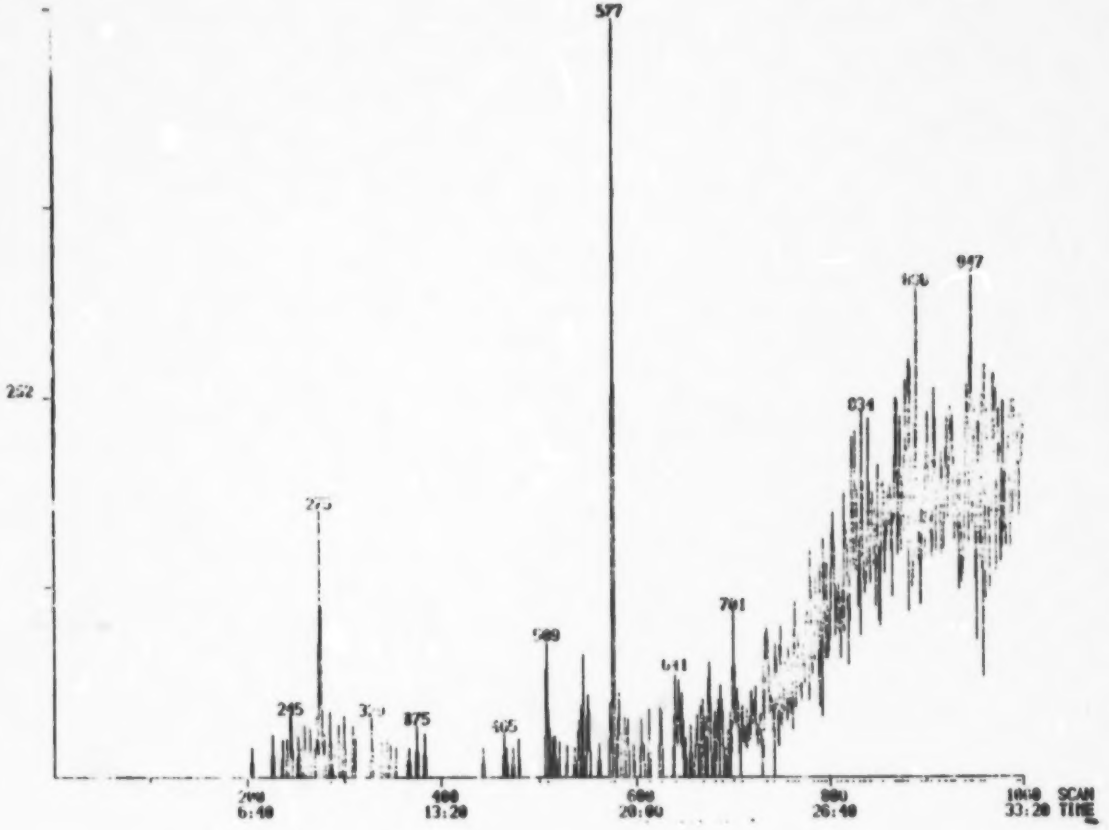

Figure 2. Mass chromatogram of m/e 252.

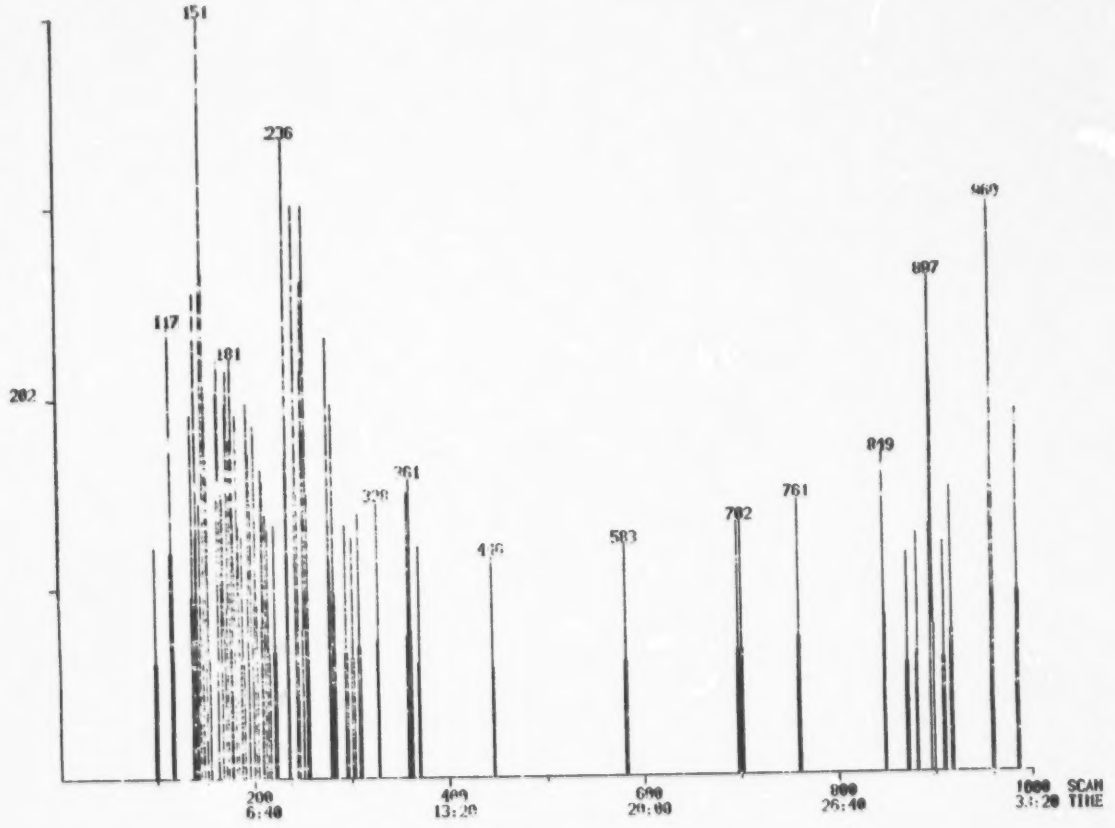

Figure 3. Mass chromatogram of m/e 202.

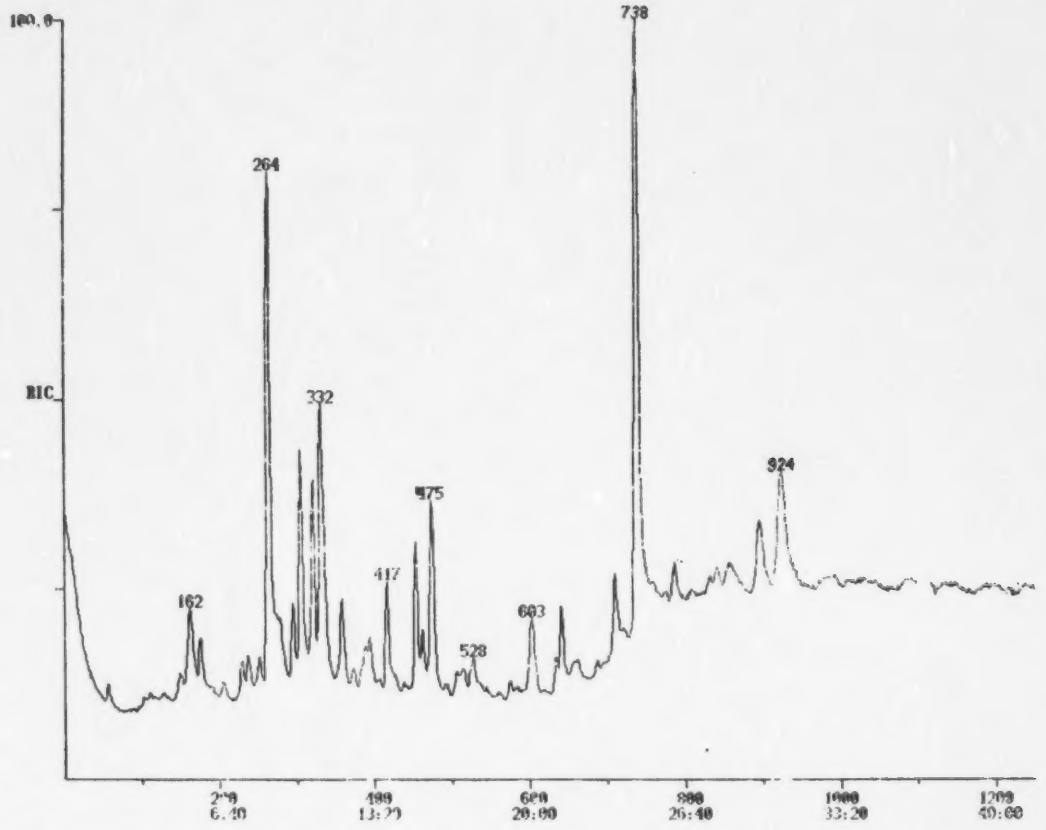

Figure 4. Total ion chromatogram of a typical coke oven sample.

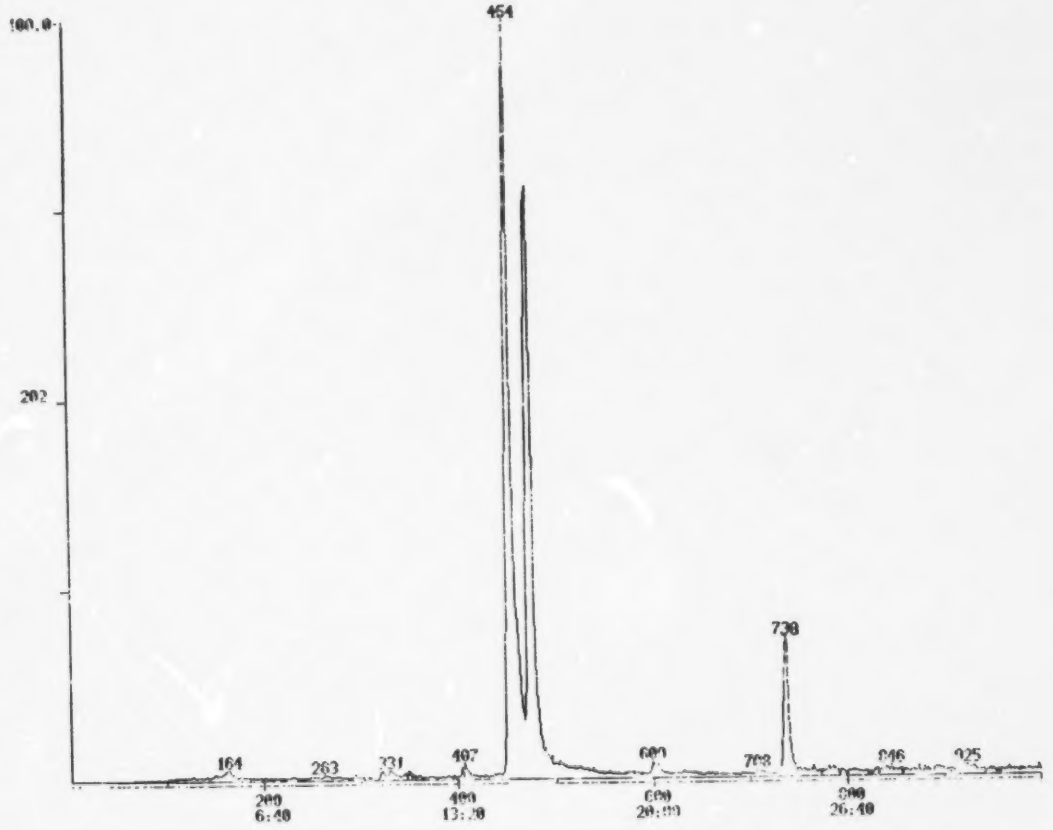

Figure 5. Mass chromatogram of m/e 202.

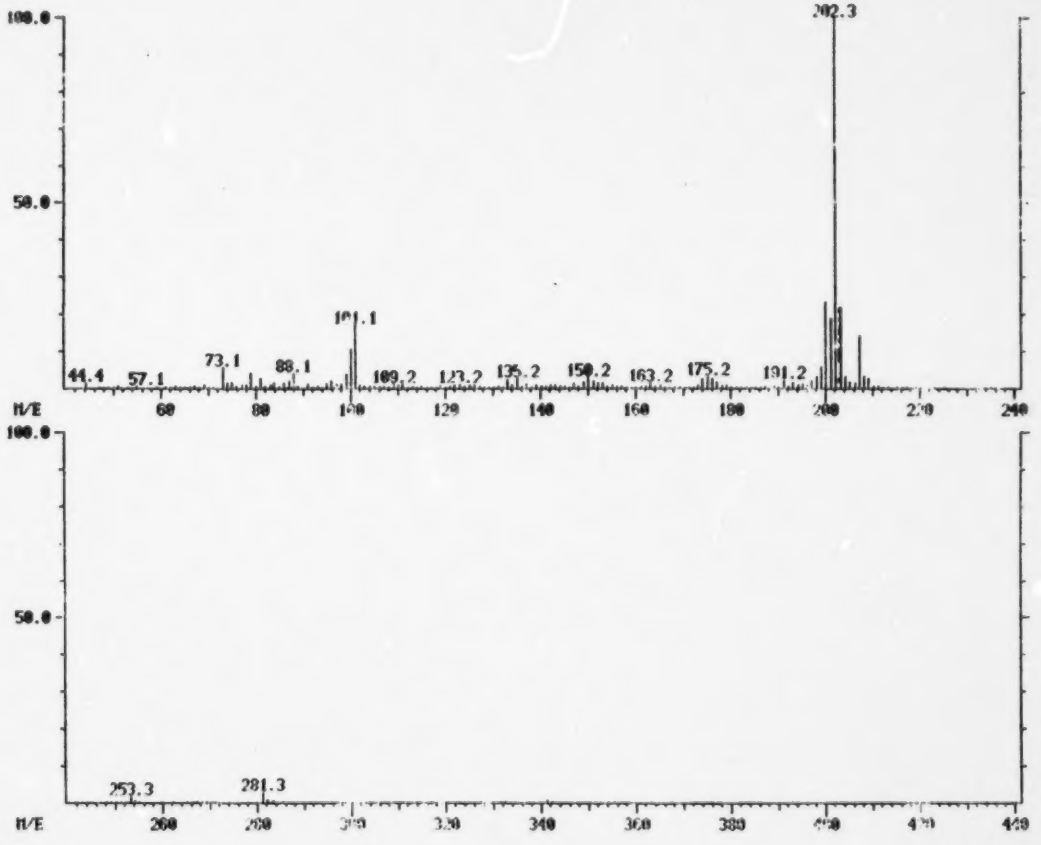

Figure 6. Mass spectrum - scan number 454.

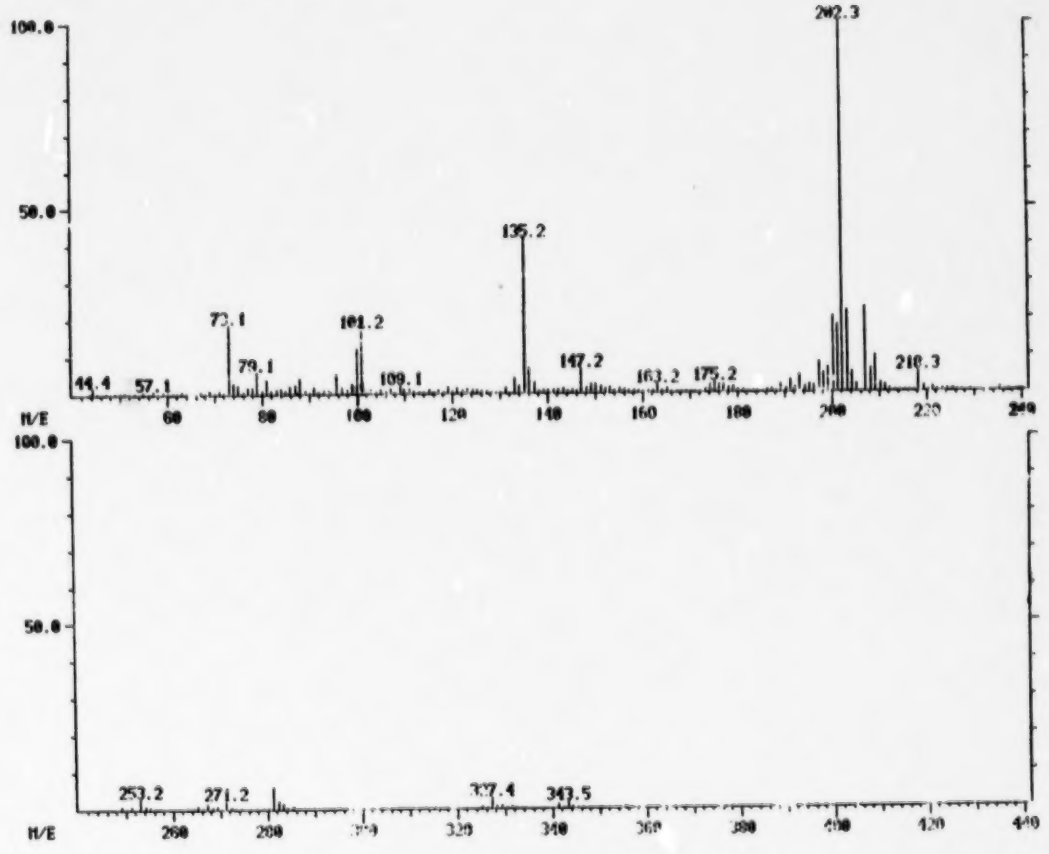

Figure 7. Mass spectrum - scan number 476.

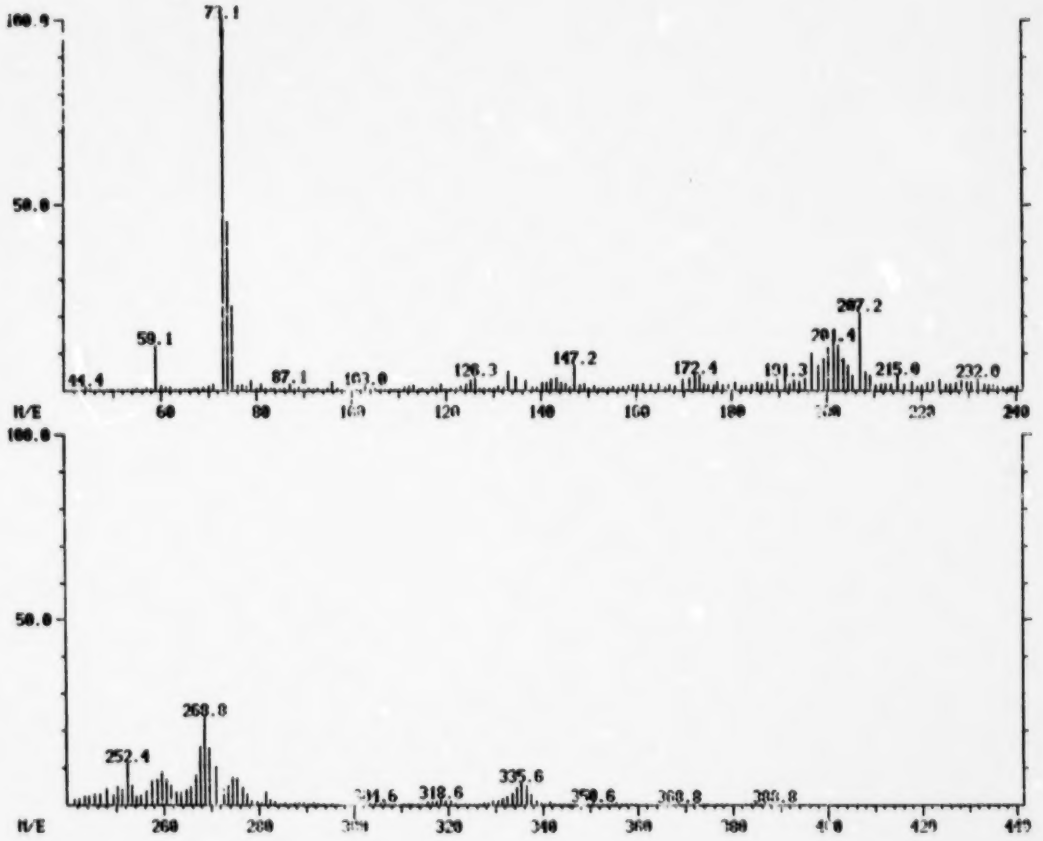

Figure 8. Mass spectrum - scan number 738.

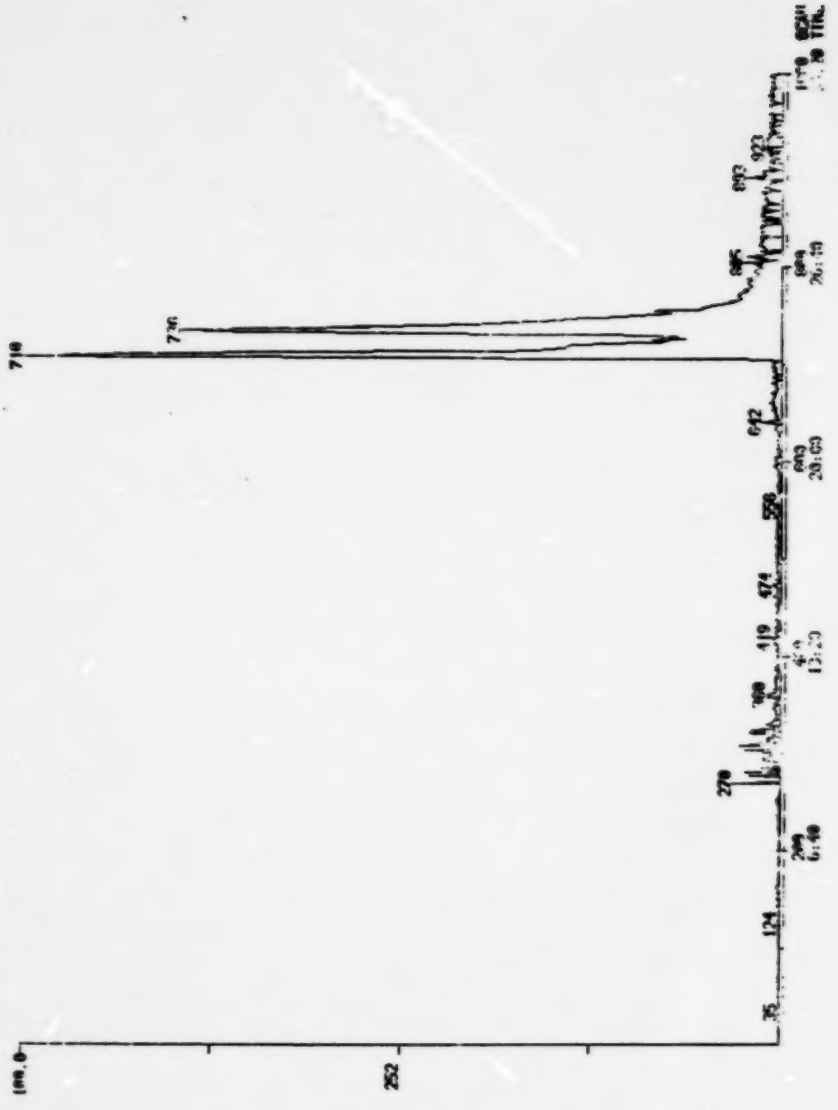

Figure 9. Mass chromatogram of m/e 252.

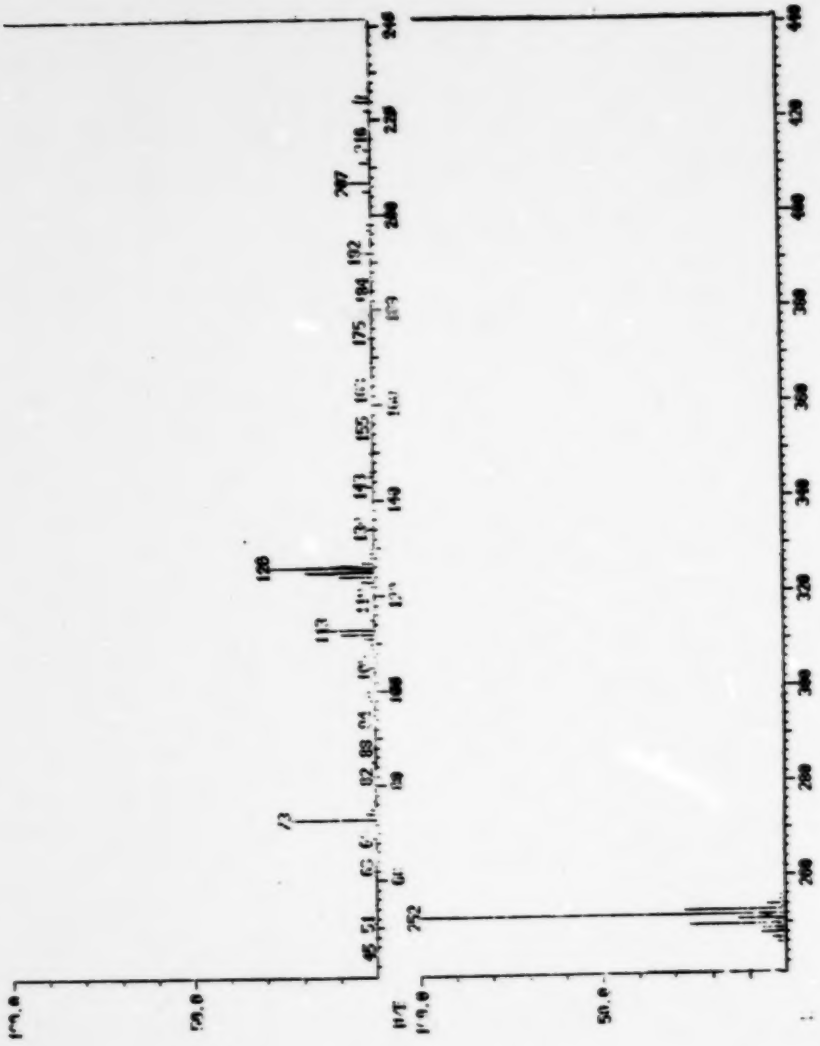

Figure 10. Mass spectrum - scan number 710.

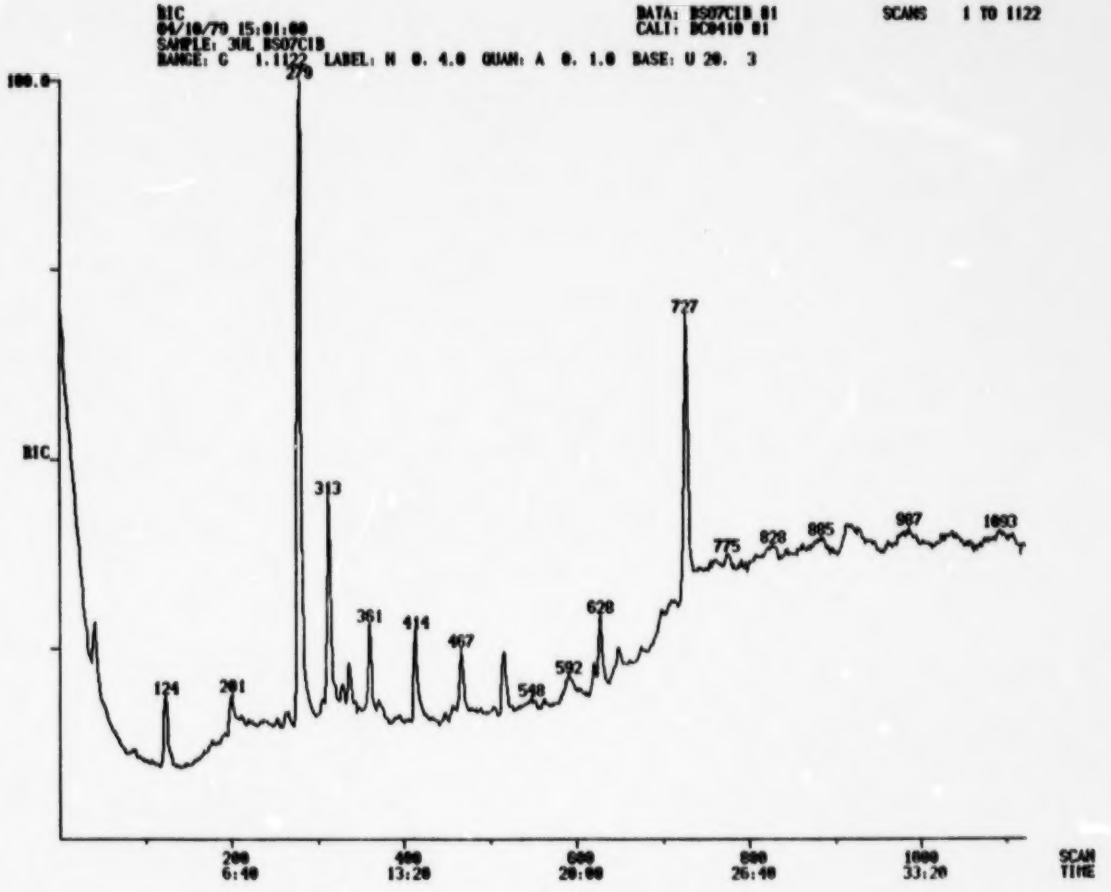

Figure 11. Total ion chromatogram of detection limit test sample.

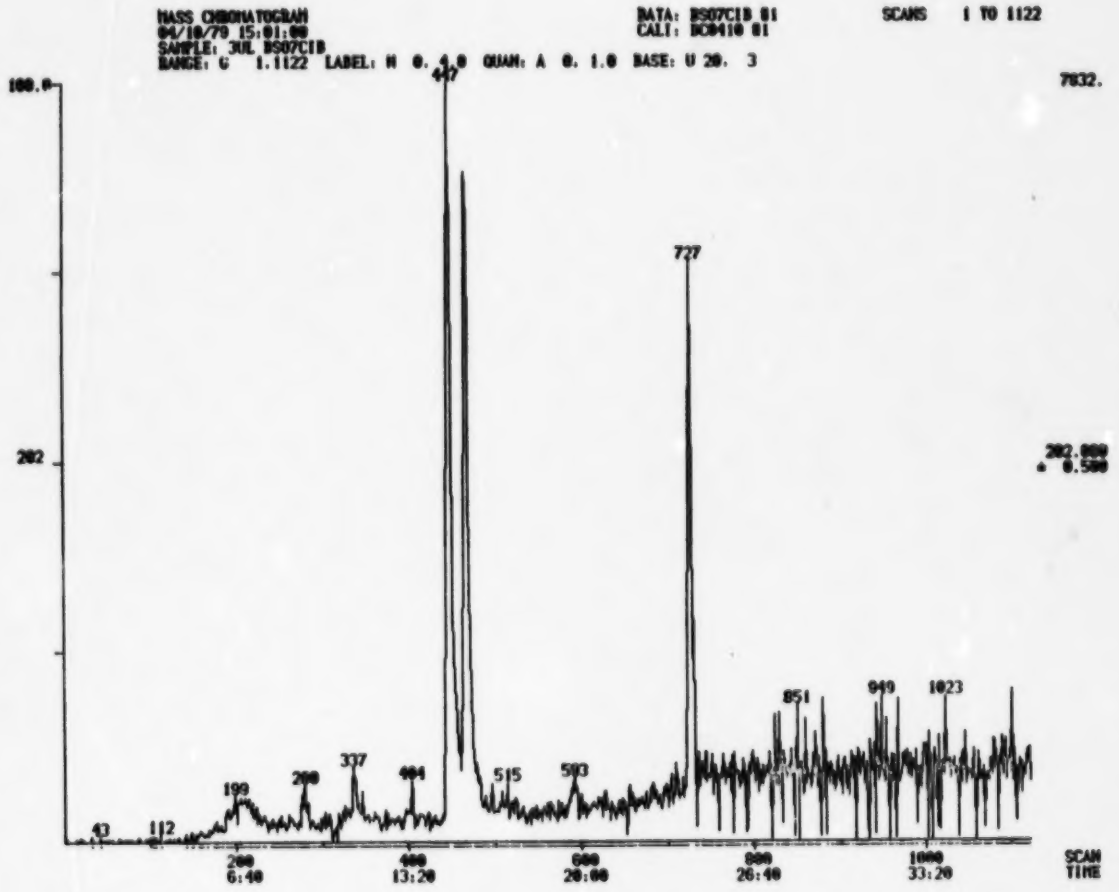

Figure 12. Mass chromatogram of m/e 202.

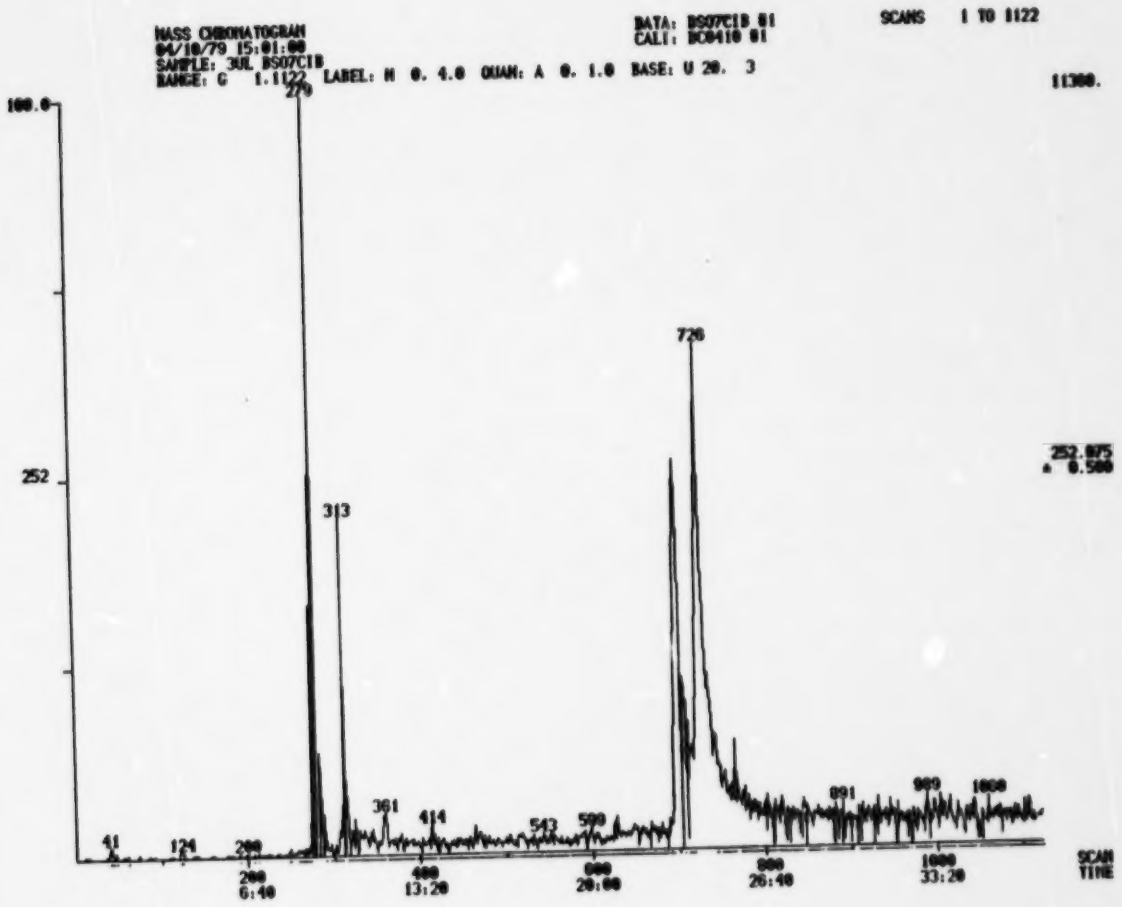

Figure 13. Mass chromatogram of m/e 252.

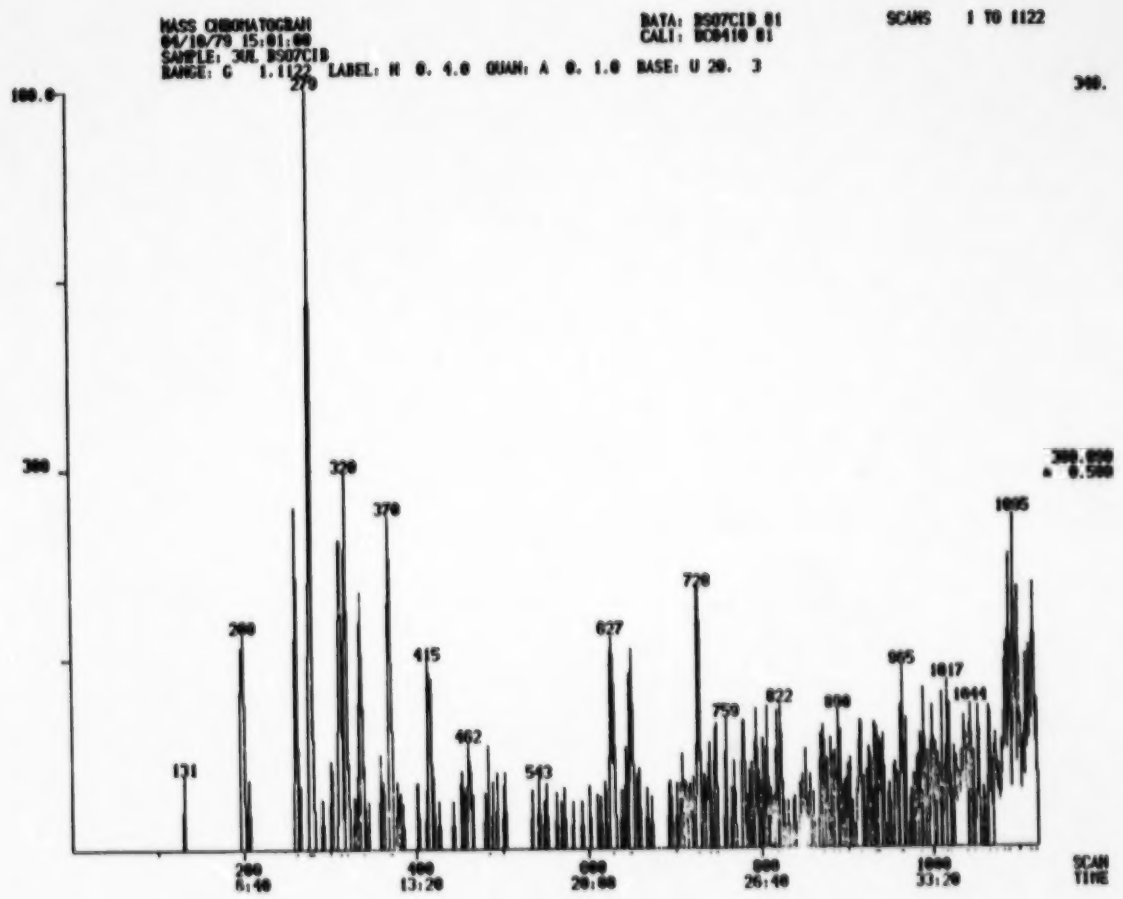

Figure 14. Mass chromatogram of m/e 300.

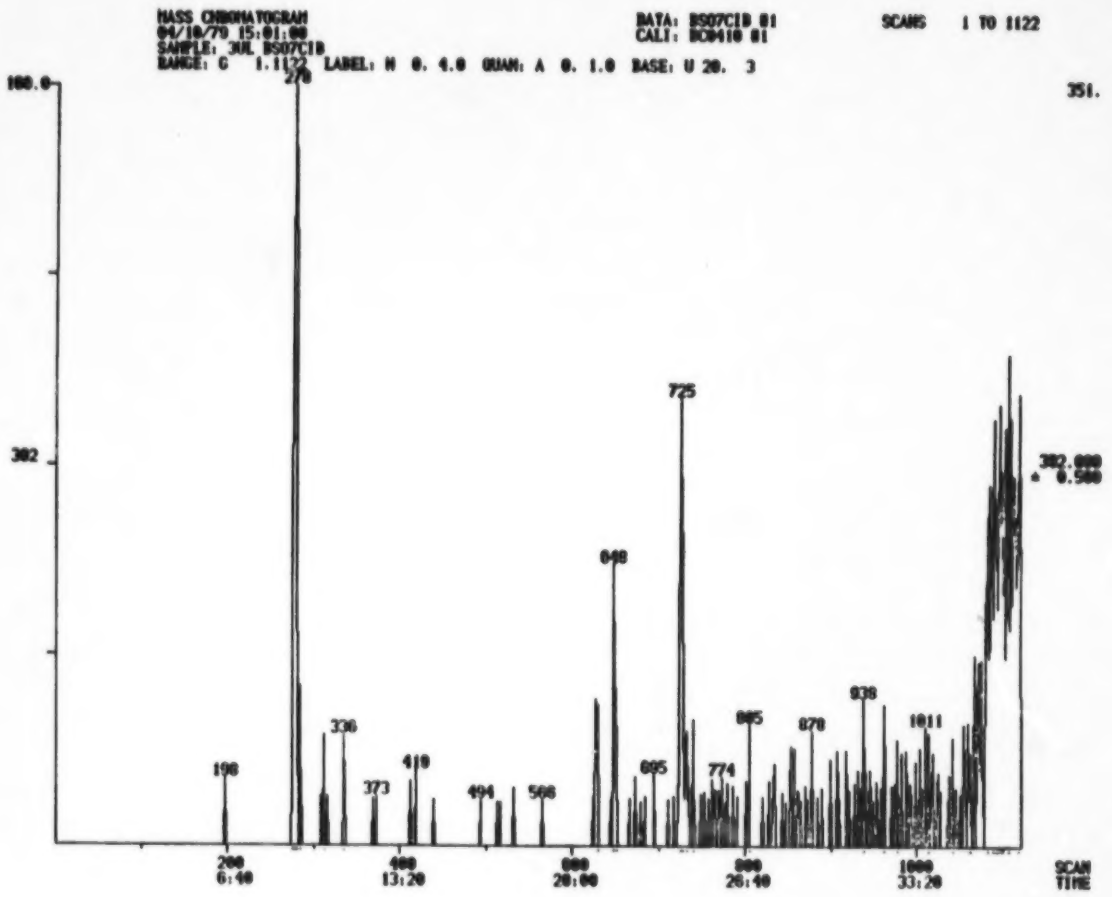

Figure 15. Mass chromatogram of m/e 302.

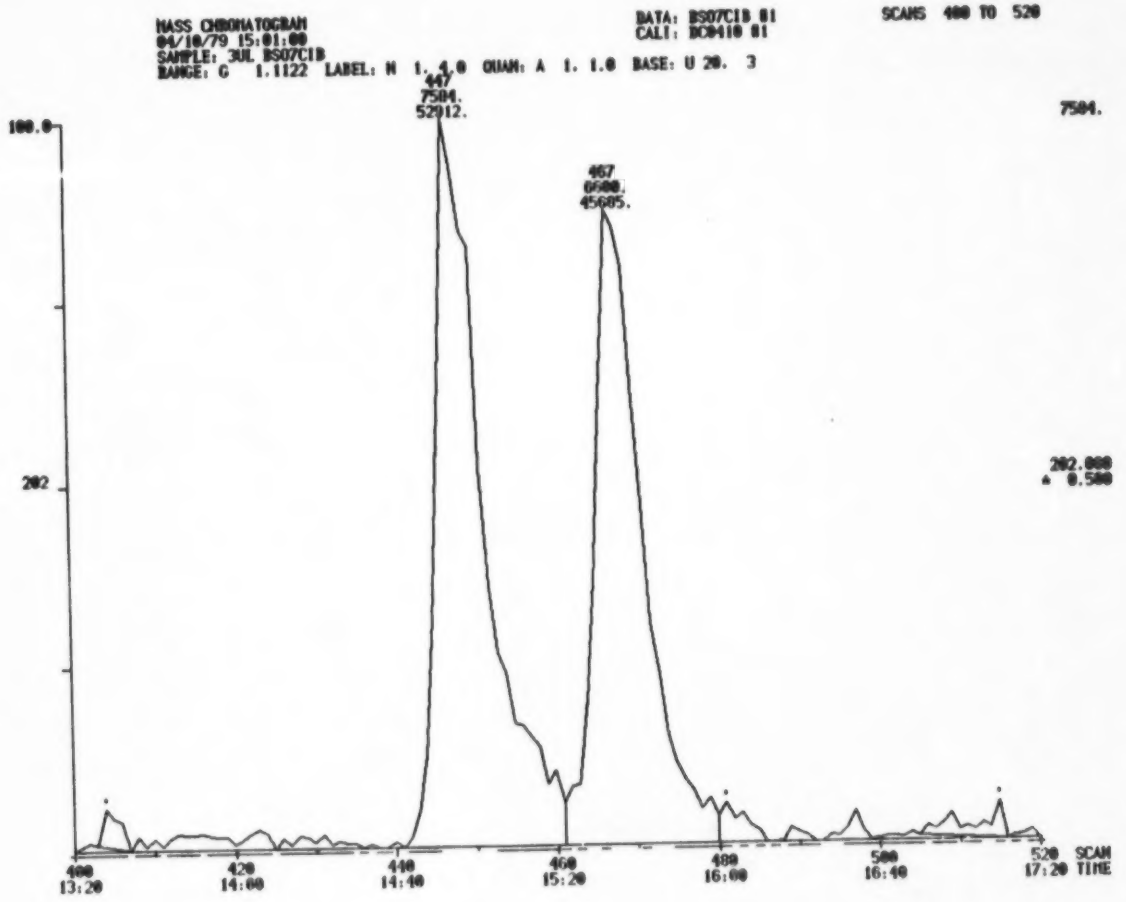

Figure 16. Mass chromatogram of m/e 202.

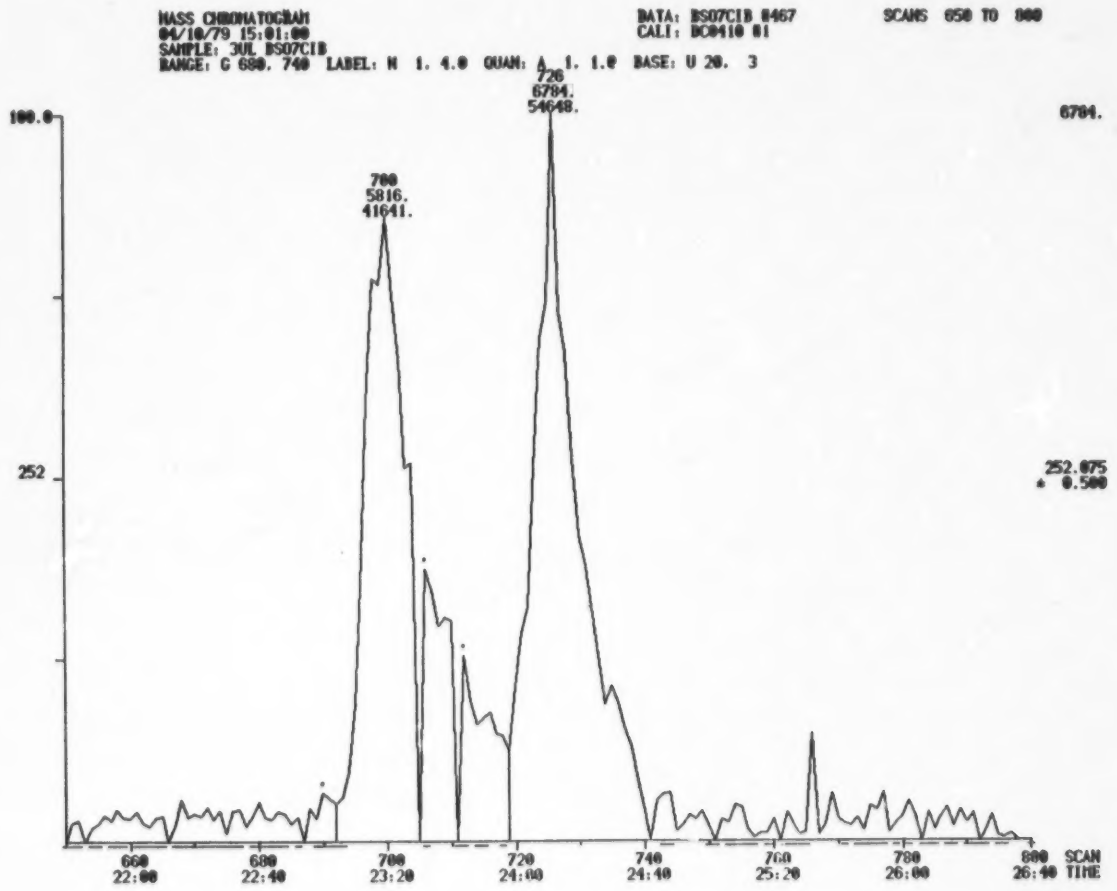

Figure 17. Mass chromatogram of m/e 252.

5.0 SUGGESTIONS FOR FUTURE STUDY

Other alternatives exist to the screening technique proposed in the Arthur D. Little, Inc. report. High performance liquid chromatography (HPLC) is a technique that can provide a semi-qualitative and quantitative analysis for PAH in a time span as short as 15 minutes. As with the gas chromatograph, the identity of the eluting components are assigned by retention time and spiking. Specific detection techniques such as ultraviolet absorption and fluorescence can be employed to screen for PAHs. The selectivity of these detectors are extremely useful when analyzing complex samples. Setting the UV or fluorescence detector at wavelengths specific for the PAH(s) provides for an enhanced response of the components of interest over other components in the sample matrix.

Figures 18 through 20 are high performance liquid chromatograms of PAH standards and a coke oven sample. Figure 18 is a standard mixture of benzene, chloronaphthalene and benzo(a)pyrene. Under these conditions, 20% water in methanol at a flow of 1 milliter per minute on a 25 cm ODS column, benzo(a)pyrene elutes in approximately 35 minutes while benzene elutes in 7 minutes. Figure 19 is the chromatogram of naphthalene, fluorene, phenanthrene and benzo(a)pyrene. In this case it takes 70 minutes for benzo(a)pyrene to elute from the column. This longer analysis time allows for better identification of components in complex mixtures. Figure 20 is a coke oven sample run under the same conditions as the standard in Figure 19. As can be seen, this provides an acceptable separation of a very complex sample. The presence of fluorene and phenanthrene can be easily distinguished.

These examples are typical of the results that can be obtained using HPLC and a UV spectophotometer detector.

Unlike nonaqueous reverse phase chromatography (NARP), conventional reverse phase high performance liquid chromatography involves the use of nonpolar chromatographic stationary phase (e.g. c-18 bonded to an inert support) and a polar solvent system (e.g. methanol/water). In the case of PAH compounds, the use of this type of system for separation of PAHs above 300 molecular weight can result in excessive analysis time. This is due in part to the limited solubility of PAHs in the solvent systems used. Substituting a less polar solvent system for the polar solvent systems

routinely used in reverse phase chromatography increases the solubility of the PAHs. Consequently, with the use of methylene chloride and acetonitrile as a solvent system, coronene (m/w 300) and decacyclene (m/w 454) can be eluted in approximately 8 and 17 minutes, respectively. Thus with NARP, components which cannot be analyzed by GC/MS can be handled easily and in a relatively short analysis time. It is recommended that HPLC be investigated as an additional method for the analysis of PAHs.

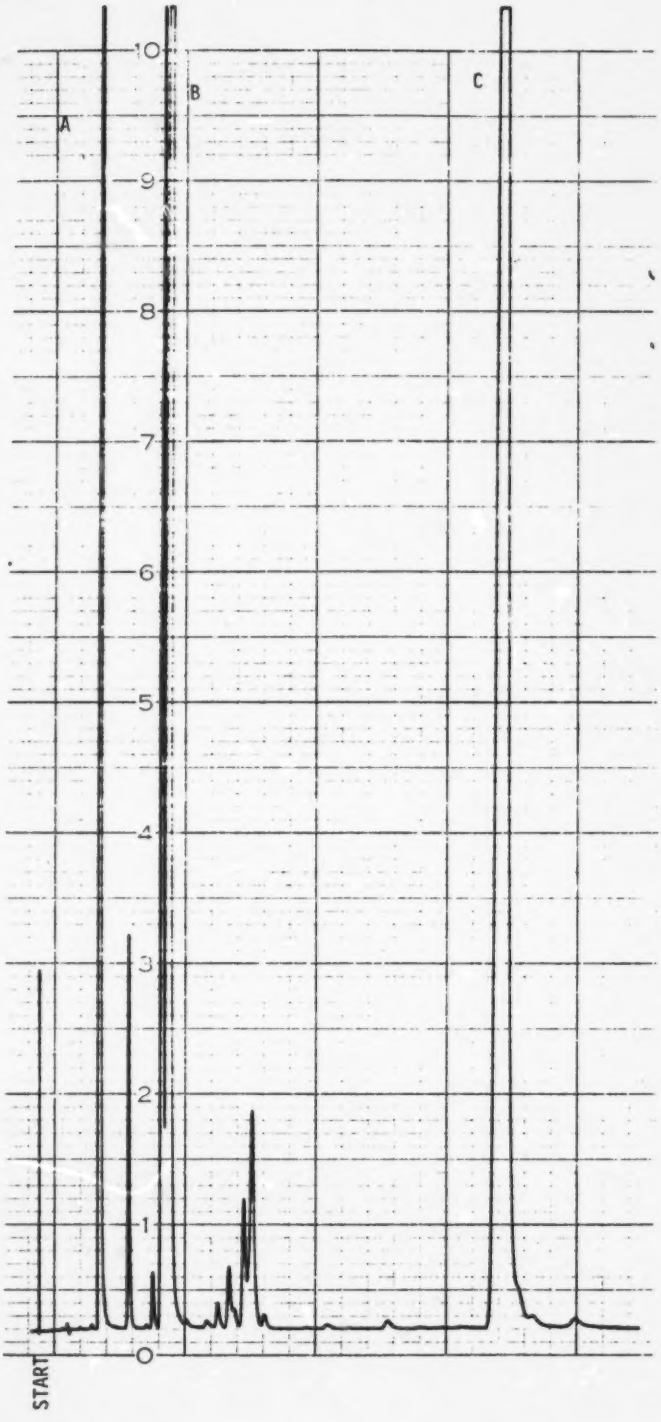

Figure 18. High performance liquid chromatogram of A. benzene, B. chlorobenzene and C. benzo(a)pyrene.

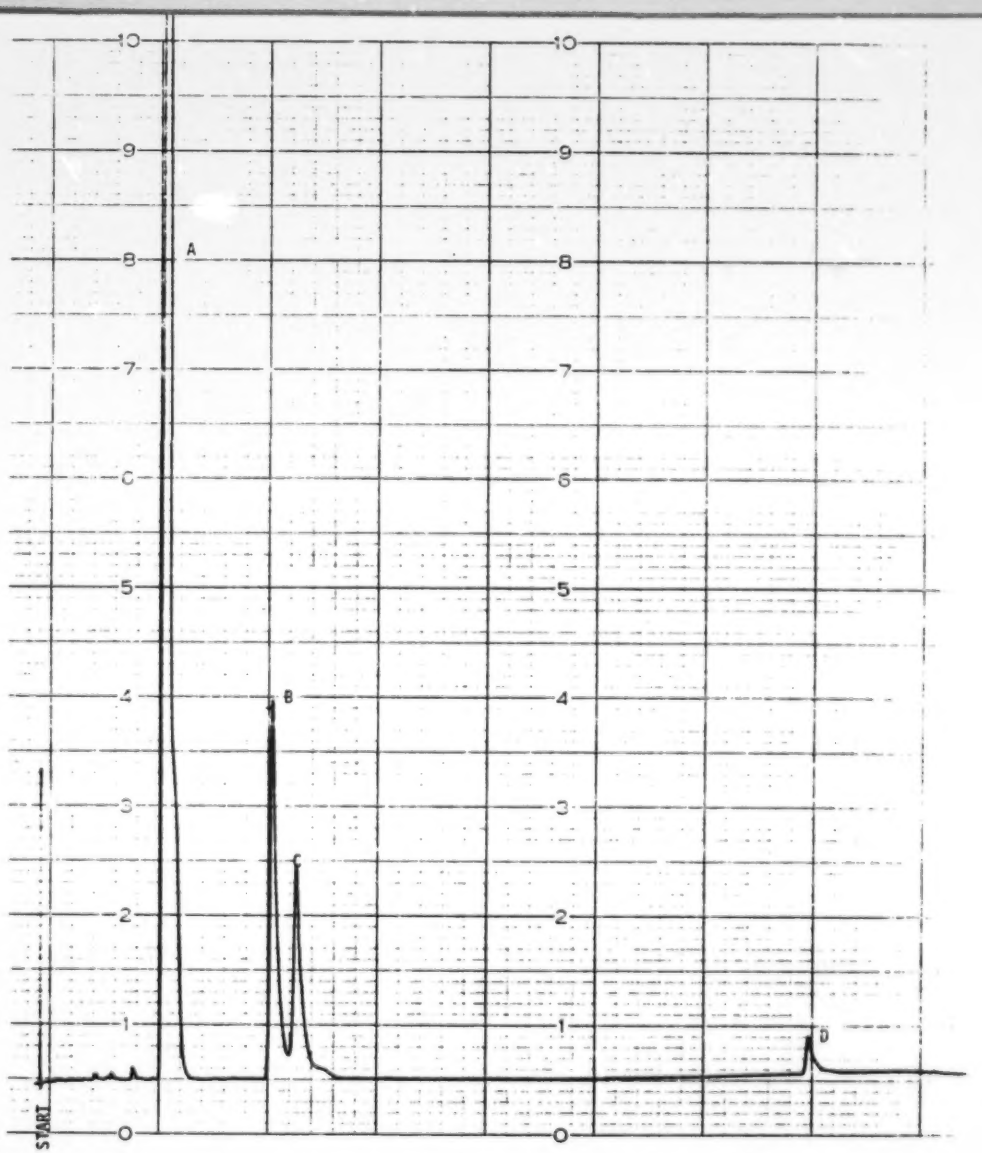

Figure 19. HPLC of A. naphthalene, B. fluorene, C. phenanthrene and D. benzo(a)pyrene.

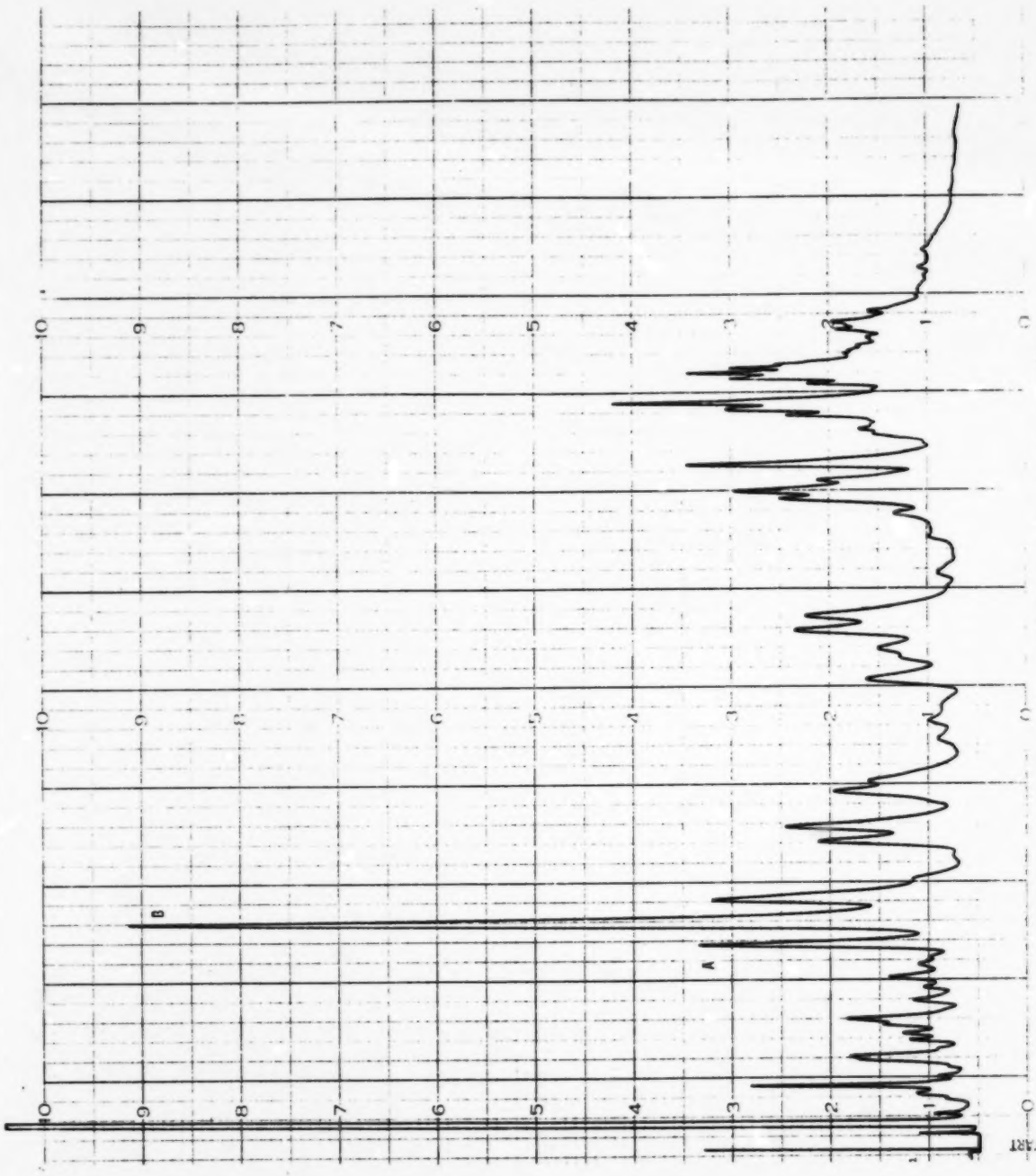

Figure 20. HPLC of coke oven sample
A. fluorene; B. phenanthrene.

6.0 CONCLUSION

It is clear from the data presented in this report that the spot test has proven acceptable as a screening tool for samples submitted for GC/MS analysis of PAHs. Two requirements were to be met by this screening procedure. One, the detection limit of the PAH spot test must be below that of the GC/MS and two, the interferences that prevent accurate results from the spot test must be defined. These issues have been addressed. That is, the detection limit of the screening technique (spot test) is below that of the accepted analysis technique (GC/MS). Also, interferences occuring from the inherent color of the sample were discussed.

It was shown that unconfirmed positives can be obtained from several sources. A unconfirmed positive result will occur when a sample contains a level of PAHs which is above the detection limit of the spot test and below the detection limit of the GC/MS. Another source of unconfirmed positives can be caused by the interference of the sample color with the fluorescence of the sample.

Another source of false positives which must not be overlooked are cases in which the fluorescence is caused by PAHs which have a molecular weight above 300. GC/MS will not prove adequate as a method of analysis. Clearly, high performance liquid chromatography must be used in cases where high molecular weight PAHs are suspected. Indeed, it may prove beneficial to analyze all samples which prove positive by the spot test by HPLC for high molecular weight PAHs and not only those which prove positive by the spot test and negative by GC/MS.

7.0 REFERENCE

1. Sensitized Fluorescence for the Detection of Polycyclic Aromatic Hydrocarbons, Report No. EPA-600/7-78-182, NTIS No. PB 287-181 IAS, September 1978.